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PROJECT CLOSING REPORT

"DEVELOPMENT OF ADVANCED MULTIZONE FACILITIES FOR MICROGRAVITY PROCESSING".

(NCC-3-191, TWIR-13)

INTRODUCTION

NASA has been interested in experimental ground based study to investigate the fundamental processes involved in phase transformation processes during growth of metallic, nonmetallic and electronic materials. Solidification, vapor growth and solution growth techniques of growing crystals are of special interest because of the inherent importance of convection in the nutrient solution. Convection enhances the mass transport through the nutrient and results in faster growth rates. However, it also alters the thermal profiles, both in the melt and the solidified portion, and thus affects the shape of the growth front. Shape of the growth front is closely related to the extent of micro and macrosegregation of solutes (dopants), which determines the overall yield and quality of the electronic material. Availability of low gravity environment of space has provided scientists a new variable to control the extent of convection and thus isolate the diffusive phenomena for their better understanding.

Most of the phase transformation studies are carried out in furnaces where the nutrient is kept hotter than the crystal seed during the growth process. The thermal profiles in-side the crucible are created by the heaters located out side. For example alloys are directionally solidified in a modified Bridgman type of directional solidification furnace, where the furnace usually consists of a hot zone and a cold zone separated by an insulated (adiabatic!) zone. The thermal gradient at the liquid-solid interface is determined by the alloy characteristics, the hot zone temperature, cold zone temperature and the width of the insulating zone. This arrangement does not allow the researcher to impose a variable and controlled thermal profile along the length of the hot zone. It is usually not possible to control the thermal profile near the liquid-solid interface. The thermal profiles therefore get established by the existing material and geometrical constraints of the experimental set up. There is a critical need to have a directional solidification type of furnace, where the temperature profiles of varying nature can be independently imposed at the liquid-solid interface or in the melt column, and then controlled during subsequent solidification processing. This would enable the researchers to investigate phenomena, such as, the effect of the nature of the interface curvature (convex, concave, or planar) on the extent of gravity driven natural convection in the melt and the resulting electronic properties, macrosegregation of solutes along the length and width of the directionally solidified ingots, the role of radiation in causing convection in semitransparent salt crystals, etc.

WORK PERFORMED UNDER THIS PROJECT:

(A) **The major effort** under this research was devoted to designing a programmable furnace which can be used to obtain thermal profiles along the length of the sample as per the demands of the scientists. The furnace did not have active cooling of the zones. Only active heating and passive cooling were utilized. Several sectors of such a furnace were fabricated and tested for their power efficiency, thermal profiles, programmability of temperatures and temperature profiles, etc. Unfortunately the concept of programable multizone furnace was later on transferred to NASA-Marshall Space Flight Center. But it is expected to derive substantial background knowledge base generated under this project. Availability of such a furnace will not only impact our understanding of the various phase transformation phenomenon on ground, but will also be an asset for a more meaningful investigation of many low gravity processes. Directional solidification can then be attempted without having to translate either the furnace or the specimen, by only controlling the temperatures in the various segments as a function of time, thus traversing the liquid-solid interface along the length of the ampoule. The same furnace can also be used to provide isothermal or constant gradient along the length of the melt column to carry out experiments involving diffusion in liquid, thermomigration (solutal diffusion because of the presence of thermal gradient) in melt and growth of electronic materials with simultaneous vapor pressure control to vary the dopant distribution and heat treatment subsequent to the crystal growth to reduce thermal stresses which are so detrimental to the crystal quality. The same furnace can also be used for studies involving zone refining of single crystal materials, by generating a thermal profile which maintains a constant temperature hot zone in the middle while keeping the rest of the specimen solid. The furnace can be used in thermally stable mode, low density liquid (nutrient) which is hotter kept at the top and cooler melt at the bottom. It can also be used to study thermally unstable mode of crystal growth, high density cooler melt on top of a hotter low density melt, with gravity pointing down.

(B) Under **second aspect** of this project support was provided to the low gravity Ostwald Ripening experiments of Dr. Peter Vorheese (North Western University) in the lead-tin alloys, currently funded by the Microgravity Science and Applications Division of the NASA. Specifically the design of the experimental facilities to provide a nearly isothermal environment for these low gravity experiments involving coarsening kinetics of lead precipitates in a lead-tin liquid two phase medium required extensive support. Lead-tin specimens, mechanically deformed and coarsened, were examined to provide controlled microstructure specimens for further coarsening.

(c) **The third aspect** of this project involved experimental directional solidification program about the extent of natural convection caused by a temperature inversion in a binary alloy melt during terrestrial directional solidification experiments. The High Temperature Directional Solidification Furnace in the Microgravity Materials Science Laboratory at the NASA Lewis Research Center was modified for this purpose. Hypoeutectic lead-tin alloys were directionally solidified using this facility, both with melt on top of the solid, and, also the solid on top of the melt. Thermocouples were imbedded into the melt column along its length to measure the in-situ thermal profiles. Thermal fluctuations and macrosegregation along the DS length were studied. Following conclusions can be drawn from this research on the directionally solidified Pb-38 wt pct. Sn, and Sn-35.5 wt pct. Pb alloys.

(1) Convection during downward growth, solid on top of the melt, produces temperature fluctuations along the length of the melt column. With increasing instability, i.e., larger Rayleigh number, these fluctuations change from cyclic (time periodic with a constant frequency), to oscillatory (time periodic with several harmonics), and finally to random. Application of a transverse magnetic field partially suppresses these flows, as if the

Rayleigh number had been decreased; the random flows tend to become oscillatory, and the oscillatory ones cyclic. For the case of the lower temperature inversion - the transverse magnetic field changed the flow, possibly from a double cell to a single cell, which resulted in oscillatory flows inducing an increase in temperature fluctuation and a decrease in the periodic frequency of those fluctuations.

(2) The convective flow stability diagrams presented by others, relating thermal Rayleigh number, aspect ratio, and convection, are confirmed and in good agreement with our results.

(3) Macrosegregation along the length of the directionally solidified specimens is produced only when the interdendritic liquid in the mushy zone convects and mixes with the bulk melt. Despite extensive convection in the bulk melt during downward growth of Pb-38 wt pct. Sn, as evidenced by the thermal fluctuations, no longitudinal macrosegregation is observed because the interdendritic melt density profile is stabilizing. Whereas, during its upward growth of the same alloy the interdendritic melt density profile is destabilizing, and extensive longitudinal macrosegregation results.